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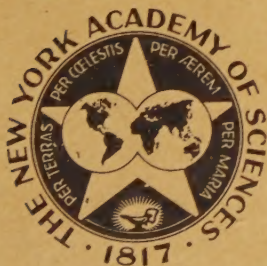
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ROY WALDO MINER

NEW AGE DETERMINATIONS BY THE LEAD METHOD

BY

J. LAURENCE KULP, GEORGE L. BATE,
AND BRUNO J. GILETTI



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Introduction

The lead method of age determination was put on quantitative basis by the classic work of Nier ^{1,2} around 1940, when he performed the necessary lead isotope analyses of radiogenic leads by mass spectrometric means and corrected for common lead contamination. Subsequent developments and an analysis of the errors involved in the method as currently used are reviewed by the authors in another publication. ³

In order to obtain a closer approximation to the true age of a mineral by the lead method, additional quantitative tests have been applied in the present research. These refinements may be enumerated as follows:

(1) The Pb ^{210} concentration in a mineral has been experimentally measured which, together with the Pb ^{206} analysis, gives an additional independent age of the sample. Specifically this age is independent of recent leaching of uranium.

(2) The amount of radon emanation from some samples under laboratory conditions was measured. This measurement provides an index of the errors of the $\text{Pb }^{206}/\text{U }^{238}$ and $\text{Pb }^{207}/\text{Pb }^{206}$ ages due to radon leakage.

(3) In samples containing common lead, choice of the proper common lead composition to use for the correction was based on availability of analyses of common lead from the same metallogenic province.

(4) The errors in the $\text{Pb }^{207}/\text{Pb }^{206}$ ages arising from uncertainties in the U ^{235} half-life and in the present ratio of $\text{U }^{235}/\text{U }^{238}$ have been found to be appreciable in the case of young minerals.

Apparatus and Method

The isotopic analyses of lead were made with a 6-inch, 60-degree, direction-focussing spectrometer of glass-metal construction. A single collector was used to receive the ion currents, which were then amplified by a vibrating reed electrometer and displayed on a Speedomax recorder. The ion-accelerating potential was held constant at approximately 2500 volts, while the magnetic field was varied to provide the desired scan. The resolution of the instrument was approximately 1 part in 250. The sample was introduced into the instrument as $\text{Pb}(\text{CH}_3)_4$, which has sufficient vapor pressure at room temperature to be used directly without heating the all-metal sample manifold, as was demonstrated by Collins. ⁴

* Lamont Geological Observatory Contribution Number 104.

TABLE 1
CHEMICAL ANALYSIS DATA

Sample No.	Mineral & Locality	%Pb	%U	%Th
K- 4	Wiseman Mine, Spruce Pine, N. C. Samaraskite	0.60 ± 01	13.06 ± 02	1.11 ± 03
K- 6	Rickards Mine, Gilpin Co., Colo. Uraninite	1.34 ± 01	47.88 ± 01	0.10 ± 01
K- 7	Contact Lake, Saskatchewan Uraninite	2.51 ± 01	19.46 ± 03	0.22 ± 02
K- 8	German & Belchin Mine, Central City, Colo. Uraninite	0.79 ± 01	64.66 ± 05	0.18 ± 01
K- 9	Belgian Congo Uraninite	6.273 ± 04	73.78 ± 02	0.016 ± 005
K-10	Belgian Congo Uraninite (Minor Alteration)	6.62 ± 02	72.51 ± 02	0.008 ± 001
K-11	Belgian Congo (Crushed & leached) Uraninite	7.52 ± 05	76.00 ± 03	0.01
K-12	Marysvale, Utah Uraninite Concentrate	0.0233 ± 001	6.35 ± 05	
K-13	Marysvale, Utah Uraninite Concentrate	0.0267 ± 005	7.70 ± 02	
K-14	Eagle Mine, Lake Athabasca, Canada Uraninite Ore	6.43 ± 03	27.95 ± 04	0.0045
K-15	Joachimsthal, Bohemia Uraninite	2.74 ± 01	55.97 ± 02	
K-16	Eldorado Mine Uraninite	1.40 ± 01	6.60 ± 01	
K-18	Eldorado Mine Uraninite	8.39 ± 05	45.85 ± 01	
K-19	Nicholson Mine, Goldfields Uraninite Ore	4.91 ± 02	44.31 ± 02	
K-20	Witwatersrand, South Africa Uraninite Concentrate	14.36 ± 11	62.025 ± 01	
K-21	Upper Huronian Iron formation Pitchblende Concentrate	3.903 ± 03	70.21 ± 07	
K-25	Sofia Mine, Boden, Germany, Pitchblende Ore	0.96 ± 01	32.97 ± 11	

This procedure permitted a short pump down time between runs with no memory effects.

The isotopic abundances were calculated from the Pb^+ spectrum with suitable corrections made for the monohydride PbH^+ . This correction amounted to about 10 per cent. The exact value was calculated from the peak at mass 209 for samples of high Pb^{208} content. A complete discussion of the mass spectra of group IV tetramethyl compounds may be found in the literature.⁵

Materials

The samples measured in this study were obtained from the Mineralogical Collection of Columbia University through the kindness of Professor Paul F. Kerr, and from the United States Atomic Energy Commission through the cooperation of Mrs. M. Mathez. The chemical analyses were done by the U.S. Atomic Energy Commission; by the New Brunswick Analytical Laboratory, New Brunswick, N.J.; and by Ledoux and Co., New York, N.Y. The lead tetramethyl preparation was carried out in this laboratory using a procedure modified after that of Collins.⁴ The isotopic analyses, Pb^{210} determinations, radon leakage measurements, and the petrographic examinations were made in this laboratory.

Results

The chemical analysis data are given in TABLE 1. The isotopic compositions of the lead extracted from the radioactive minerals are listed in TABLE 2. In order to obtain the most precise correction for common lead, care was taken to choose the best available common lead data for sites as close as possible to the deposit which produced the radioactive mineral. The locality of the common lead used and the final percentages of radiogenic 206, 207, and 208 are given in TABLE 3. The ages for the ratios 206/238, 207/235, 208/232, 207/206 were computed with the aid of simplified nomographs published elsewhere.³ These results are listed in TABLE 4 along with the 206/210 ages. The latter are measured by alpha counting the Po^{210} daughter of Pb^{210} . The Pb^{206} concentration is determined by mass spectrometric analysis. The theory and technique used in the Pb^{210} method is described in detail in another paper.⁶ It suffices here to point out that since Pb^{210} is below radon in the U^{238} decay chain, the ratio 206/210 will be independent of radon leakage if the leakage has been reasonably constant throughout the age of the mineral. Further, if uranium leaching takes place late in the history of the mineral, the lead will be essentially unaltered and the 206/210 age will be unaffected. Not only is the 206/210 age easiest to measure, but it is very insensitive to changes in external conditions.

TABLE 2
ISOTOPIC DATA

Sample No.	Locality	204	206	207	208	Mineral
K-4	Wiseman Mine Spruce Pine, N. C.	0.02	91.2 ± 2	5.60 ± 08	3.18 ± 07	Samaraskite
K-6	Rickards Mine Gilpin Co., Colo.	0.97 ± 01	44.88 ± 20	16.63 ± 05	37.52 ± 10	Pitchblende
K-8	German & Belchin Mine Central City, Colo.	0.57 ± 01	65.7 ± 1	11.78 ± 04	21.97 ± 20	Pitchblende
K-9	Katanga Belgian Congo	0.01	93.6 ± 03	5.79 ± 08	0.32 ± 02	Ureninite (hard and shiny)
K-10	Katanga Belgian Congo	0.01	94.0 ± 05	5.90 ± 09	0.06 ± 02	Uraninite (minor alteration)
K-11	Katanga Belgian Congo	0.01	94.0 ± 03	5.93 ± 07	0.07 ± 02	Uraninite (acid leached)
K-7	Contact Lake N. W. T.	0.01	92.3 ± 2	7.50 ± 20	0.27 ± 03	Pitchblende
K-14	Eagle Mine (200-300') Lake Athabasca	0.01	91.20 ± 15	8.61 ± 20	0.18 ± 06	Pitchblende
K-16	Eldorado Mine (151 Stope)	0.12 ± 01	86.4 ± 1	9.27 ± 15	4.26 ± 10	Pitchblende Ore
K-18	Eldorado Mine (913 Stope) Great Bear Lake	0.24 ± 01	82.6 ± 1	9.66 ± 05	7.52 ± 04	Pitchblende
K-19	Nicholson Mine (No. 2 shaft) (Goldfields dump)	0.02	92.44 ± 03	7.25 ± 10	0.32 ± 02	Pitchblende Ore
K-15	Joachimsthal	0.62 ± 02	60.81 ± 06	13.10 ± 04	25.45 ± 04	Pitchblende (clean)
K-20	Witwatersrand	0.1	85.4 ± 2	11.38 ± 06	3.15 ± 10	Pitchblende (concentrate)
K-21	Upper Huronian Iron Formation Mich.	0.05	93.25 ± 05	6.00 ± 08	0.75 ± 05	Pitchblende (concentrate)
K-23	Sofia Mine Baden, Germany	0.71 ± 02	61.2 ± 4	12.64 ± 15	25.4 ± 2	Pitchblende Ore
K-12, 13, 14	K-12, 13 Marysvale, Utah 13.6%	0.105 ± 02	85.80 ± 08	0.72 ± 08	4.22 ± 04	

In addition to the samples studied in this laboratory, those of other workers, for which isotopic data are available are also listed for purposes of comparison. Finally, TABLE 5 compares the ages of 10 representative samples with and without the correction for the experimentally measured radon leakage at room temperature. A recent study of the radon leakage of radioactive minerals⁷ shows that the radon leakage increases with temperature. Therefore, the integrated radon leakage must in all cases be greater than that shown in TABLE 5.

It should be observed from TABLE 4 that the agreement between different laboratories on different specimens from the same localities is quite good, indicating that the anomalies are not derived from errors in the isotopic or chemical measurements.

Discussion

With reference to the Spruce Pine samarskite (K-4), it is seen that the internal agreement of all of the ages is good except for the 207/206 age, which is usually unreasonably high for post-Cambrian minerals. It is known¹³ that the Spruce Pine district was subjected to a second regional metamorphism about 100 million years after these radioactive minerals were formed. Although the radon leakage of this samarskite at room temperature proved to be less than 0.1 per cent, the large increase in temperature over a considerable period of time may explain the spread

TABLE 3
CORRECTED PER CENT RADIOGENIC LEAD

Sample	Common Lead Locality	% Radiogenic Lead		
		206	207	208
K- 4	- - -	91.2±.2	5.60±.08	3.18±.07
K- 6	¹ Av. Colorado Plateau	26.1±.5	1.30±.33	---
K- 8	Av. Colorado Plateau	54.6±.3	2.84±.20	---
K- 9	² Kengere, Belgian Congo	93.6±.03	5.79±.08	---
K-10	Kengere, Belgian Congo	94.0±.05	5.88±.09	---
K-11	Kengere, Belgian Congo	94.0±.03	5.90±.07	---
K- 7	³ Great Bear Lake	92.2±.2	7.36±.20	---
K-14	Great Bear Lake	91.2±.2	8.53±.20	---
K-16	Great Bear Lake	84.5±.2	7.4±.2	---
K-18	Great Bear Lake	79.2±.2	6.4±.1	---
K-19	Great Bear Lake	92.3±.1	7.1±.1	---
K-15	⁴ Av. Southern Germany	48.7±.2	2.6±.2	---
K-20	⁵ Witwatersrand	80.7±.2	9.48±.07	---
K-21	⁵ Michigan	92.9±.2	5.67±.12	---
K-23	⁴ Av. Southern Germany	50.8±.5	2.15±.20	---
K-12, 13, 14	⁵ Marysville, Utah	37.2±.11*	4.3±1.6*	---

¹Stieff *et al.*⁹

²Collins *et al.*⁸

³Nier,¹⁰

⁴Nier,¹⁰ and Vinogradov *et al.*¹¹

⁵Kulp *et al.*¹²

*Per cent radiogenic 206 and 207 from the K-12, 13 part of the sample only.

TABLE 4
AGES FROM VARIOUS ISOTOPIC RATIOS

Sample*	Locality	208/232	207/206	206/238	207/235	206/210
K-4	Spruce Pine, N. C.	354±35	699±35	312±6	362±10	325±15
C'	Spruce Pine, N. C.	-----	370±40	-----	-----	-----
K-6	Gilpin Co., Colo.	-----	220±500	55±2	58±10	61±3
K-8	Central City, Colo.	-----	250±250	51±1	56±5	58±3
K-25	Gilpin Co., Colo.	-----	280±350	59±1	64±10	-----
N-24	Woods Mine, Colo.	-----	183±300	56±1	59±11	-----
K-9	Katanga (clean)	-----	676±25	576±4	598±8	650±15
K-10	Katanga (minor alteration)	-----	703±30	645±10	617±15	645±15
K-11	Katanga (acid leached)	-----	705±30	668±7	677±7	650±15
N-2	Katanga	-----	630±14	615±4	609±6	-----
N-6	Katanga	-----	632±13	567±5	577±8	-----
C'	Katanga	-----	612 to 655	-----	-----	-----
K-15	Joachimsthal	-----	425±200	179±3	197±15	-----
N-1	Joachimsthal	-----	165±180	226±5	221±22	206±10
K-16	Eldorado	-----	1407±50	1230±10	1286±20	-----
K-18	Eldorado	-----	1251±45	1015±5	1088±14	1208±40
N-10	Great Bear Lake	-----	1397±20	1215±10	1271±15	1105±30
C-14	Eldorado	-----	1430-1330	-----	-----	-----
K-21	Upper Huronian	-----	650±35	384±5	421±6	-----
K-23	Baden, Germany	-----	-----	111±2	97±13	-----
K-14	Eagle Mine, Lake Athabasca	-----	1523±50	1425±10	1450±20	1448±35
C'	Eagle Mine, Lake Athabasca	-----	1100 to 1700	-----	-----	-----
K-19	Nicholson Mine No. 2, Goldfields	-----	1141±12	730±8	843±10	808±25
C'	Nicholson Mine No. 2, Goldfields	-----	1160±30	-----	-----	-----
K-7	Contact Lake (N.W. Terr.) Can.	-----	1212±60	840±5	942±24	864±35
K-12, 13	Marysvale, Utah	-----	-----	9.8±1.2	24±10	-----
K-20	Witwatersrand	-----	1960±40	1282±15	1562±20	1290±40
C'	Witwatersrand	-----	2070±100	-----	-----	-----

*K - Determination made at the Lamont Geological Observatory.

N - Nier. 1,2

C - Collins. 8

C' - Collins unpublished data.

in the ages. If there is an integrated radon leakage of about 14 per cent, the 206/238 age would be brought up to about 350 million years, and the 207/206 would be reduced to about 350 million years. The most probable age for the first major regional metamorphism in the Spruce Pine District is 345 ± 10 million years, which produced the pegmatites and the uranium minerals. This would date the climax of this regional metamorphic cycle in the southern Appalachians at approximately the Ordovician-Silurian boundary.

The samples from the Central City district, Gilpin Co., Colo., are all in excellent agreement. As usual, the 206/238 age is low and the error in the 207/206 is too large to obtain a usable age from this ratio. Of particular interest is the agreement of the 207/235 and 206/210 ages. Note that the error is small even at these recent ages. The best age for deposit would now appear to be 59 ± 2 million years.

The Belgian Congo specimens are also in broad agreement. The radon leakage was low in these specimens (TABLE 5) and the 207/206 ages are not exorbitantly high. The three samples K-9, K-10, and K-11 provide an interesting study. K-9 was an excellent sample. K-10 showed minor alteration with evidence of minor uranium leaching. K-11 was leached with concentrated HCl for 15 minutes until all of the yellow alteration products were removed. This clearly removed uranium from the mineral. The trend of the 207/235 and 206/238 ages from K-9 to K-10 to K-11 show the likely effects of leaching. Since the violent acid treatment only increased the 206/238 by a few per cent it appears that this effect on age determinations has been greatly overrated. If uranium or lead are removed from a mineral in its very recent history, the lead-lead ratios will not be appreciably affected by leaching. Note the striking constancy of the 206/210 ages for K-9, K-10, and K-11. Further work is being done on the absolute calibration of the alpha counters for the Po ^{210} measurement which may cause revision of 5 to 10 per cent in absolute ages derived from the 206/210 ratios.

The agreement between the new data and that of Nier on the Joachimsthal locality is quite good. Again note the excellent correlation of the 207/235 and 206/210 ages. The most probable age for this deposit appears now to be 210 ± 10 million years.

Similar agreement is obtained for the Great Bear Lake samples K-16 and N-10. The K-18 sample appears to be different in age by about 150 million years. Further study is required on this problem.

Uranium mineralization in the Upper Huronian of Michigan appears to have taken place about 400 ± 20 million years ago, while that from the Sofia Mine, Baden, Germany, is close to 100 ± 10 million years.

TABLE 5
EFFECT OF CORRECTION FOR ROOM TEMPERATURE RADON LEAKAGE
ON
AGES FOR VARIOUS ISOTOPIC RATIOS

Sample	Locality	% Radon leakage measured at room temp.	207/206	206/238	207/235	210/206
K- 4	Samarskite Mitchell Co., N. C.	0.025 \pm 0.001 U	699 \pm 35 699 \pm 35	312 \pm 6 312 \pm 6	362 \pm 10 -----	325 \pm 15 -----
K- 6	Gilpin Co., Colo.	2.46 \pm 0.22 U C	220 \pm 500 167 \pm 500	55 \pm 2 56 \pm 2	58 \pm 10 -----	61 \pm 3 -----
K- 7	Pitchblende Contact Lake, Canada	7.37 \pm 0.06 U C	1212 \pm 60 1056 \pm 60	840 \pm 5 902 \pm 5	942 \pm 24 -----	864 \pm 35 -----
K- 8	Central City, Colo.	8.62 \pm 0.05 U C	250 \pm 250 34 \pm 250	51 \pm 1 55 \pm 1	56 \pm 5 -----	58 \pm 3 -----
K- 9	Katanga, Belgian Congo	1.55 \pm 0.05 U C	676 \pm 25 638 \pm 25	576 \pm 4 585 \pm 4	598 \pm 8 -----	650 \pm 15 -----
K-11	Katanga, Belgian Congo	1.36 \pm 0.02 U C	705 \pm 30 679 \pm 30	668 \pm 7 677 \pm 7	677 \pm 7 -----	650 \pm 15 -----
K-13	Marysvale, Utah	7.63 \pm 0.55 U C	----- -----	9.8 \pm 1.2 10.5 \pm 1.2	24 \pm 10 -----	----- -----
K-14	Eagle Mine, L. Athabasca, Canada	0.064 \pm 0.001 U C	1523 \pm 50 1522 \pm 50	1425 \pm 10 1425 \pm 10	1450 \pm 20 -----	1448 \pm 35 -----
K-18	Eldorado Mine Great Bear Lake, Canada	0.35 \pm 0.09 U C	1251 \pm 45 1244 \pm 45	1015 \pm 5 1019 \pm 5	1088 \pm 14 -----	1105 \pm 30 -----
K-19	Nicholson Mine No. 2, Goldfields	2.22 \pm 26 U C	1141 \pm 12 1098 \pm 8	730 \pm 8 746 \pm 8	828 \pm 10 -----	----- -----

The Athabasca area samples showed considerable spread in ages although individual deposits are self-consistent. Note the excellent agreement again between the 207/235 and 206/210 ages for K-14. The radon leakage at room temperature (TABLE 5) is inadequate to change the ages noticeably, but increased temperature may provide an integrated leakage correction of a few per cent which could account for the observed discrepancies. The K-19 ages show a much wider spread, but K-19 also has a much larger radon leakage at room temperature (TABLE 5). If the integrated leakage approached 10 per cent, the agreement would be complete. The high value of leakage for the Contact Lake sample reduces the spread for the K-7 ages appreciably (TABLE 5).

It is noteworthy that in no case does the measured radon leakage cause an "overcorrection" for the 206/238 age. Furthermore, the samples showing the greatest per cent discrepancy in the 206/238 age have the highest radon leakages at room temperature.

The Marysvale, Utah, sample was so small that it had to be diluted with a known quantity of radiogenic lead of known isotopic composition in order to prepare the $\text{Pb}(\text{CH}_3)_4$. K-14 was chosen because of its very low common lead content. The total lead present in the sample was very small, and the common lead was about 60 per cent, so that the net 207 content was small and involved a large error. The result is that the 207/235 age was almost useless. In this case, the 206/238 age is the most reliable. The Marysvale ore is very fine and has a radon leakage at room temperature of 7.6 per cent. Since this mineral was emplaced very recently under epithermal conditions, it probably has not existed for an appreciable part of its history above room temperature. Further measurements must be made in this interesting occurrence, which records the youngest known uranium mineralization. The best age at present is 10 ± 2 million years, taking into account the radon leakage.

The last sample is uraninite associated with the gold ore of the Witwatersrand, South Africa. The isotopic analysis of galena from the same mine provided an excellent base for the common lead correction. Although the 207/206 age agrees with that of Collins, the other isotopic ages do not agree internally. This is a clear case of lead loss sometime during the history of the mineral. Further study on this locality is required.

Conclusions

Sixteen new age determinations by the various isotopic ratios of the lead method have been made. The development of the Pb^{210} method is a major advance in the program of absolute age determination. It appears highly desirable to measure the radon leakage from radioactive minerals subject to age determination by the lead method. For rocks older than one

billion years the 207/206 age is best. For intermediate ages, the most reliable ages are given by the 206/210 and 207/235 ratios. For very young minerals the 206/238 age is most probable and the 207/206 of little value.

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